THE FERTILIZER VALUE OF ACTIVATED SLUDGE

BY

WILLIAM DURRELL HATFIELD
B. S. Illinois College, 1914
M. S. University of Illinois, 1916

THESIS

Submitted in Partial Fulfillment of the Requirments for the Degree of

DOCTOR OF PHILOSOPHY

IN CHEMISTRY

IN

THE GRADUATE SCHOOL

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THE FERTILIZER VALUE OF ACTIVATED SLUDGE.

By William Durrell Hatfield.

INTRODUCTION.

In the early part of the nineteenth century sewage was disposed of by discharging it into the nearest body of water. This practice soon polluted the small rivers of England until they became "boiling stinking masses." Interest in sanitary legislation in England began in 1842 when the Poor Law Commission reported that all sewage should be run on farm land. Six years later in 1848 the Public Health Act was enacted to prevent rivers and other receivers of sewage from becoming offensive to the eye and nose. These two acts were equally unsuccessful since they were not enforced. After a severe cholera epidemic, the Nuisance Removal Act, passed in 1855, marked the first really serious legislation. It was followed by the appointment of the Royal Sewage Commission in 1857, the Royal Commission on River Pollution in 1865, and the Second Royal Commission on River Pollution in 1868, to study methods of avoiding nuisance caused by dumping putrescible matter in rivers.

The history of the sanitary awakening of the authorities in Europe and America would take up too much space to be discussed here, tho it would make most interesting reading. The great amount of experimental work going on in our larger cities at the present time is evidence of the importance attached to safeguarding our streams from gross pollution.

Pollution of a stream by sewage is caused by the introduction of more suspended and colloidal organic matter than it is able to handle by its natural tendencies toward self-purification. Therefore, the best method of sewage disposal, in a particular case, is the one which so reduces the quantities of the suspended and colloidal matter that the treated sewage can be handled by the receiving body of water without overtaxing its natural purification power. The presence of this material gives ries to a large accumulation of sludge, which in the case of domestic sewage is made up of the insoluble fecal matter, paper, insoluble

material from sinks, laundries, and baths, and the grit and debris from the street washings, if allowed to enter the sanitary sewers. In case the sewage is contaminated with industrial waste many other types of solids will be present, depending on the kind of wastes. The dried sludge obtained by plain sedimentation will usually be from one-half a ton to a ton per million gallons, according to the amount of dilution water used. In Europe, the amount will usually be from 2 to 3 tons per million gallons, because less water is used for dilution. Sludge as it is removed from the sewage purification tanks is in a semi-liquid state containing from 70 to 90 per cent water, part of which must be removed before it can be handled for disposal. In some cities it may be collected in boats and dumped into the sea without removing any water, but usually it must be dried on sand beds or filter-pressed to lower the moisture content to 60 or 80 per cent, thus reducing the volume 50 to 80 per cent. This dried sludge may be disposed of by dumping, trenching, lagooning, or destructive distillation; in a few cases, after further drying, the fats are extracted, and the residue is used as a fertilizer.[10] Chemical analysis of sludges suggests their value as fertilizers, but in few instances have they been sold for this purpose.

The supply and demand of nitrogen in the United States in 1914 and 1916[17] indicated that in 1918 the demand would be 309,000 tons of nitrogen, while the prospective supply, in case of war, would be 77,000 tons. Thus the demand in 1918 is four times as great as the supply. Even before the war the importance of nitrogen fixation was recognized by the United States Government and an appropriation of $20,000,000 was made for a government nitrate plant. This appropriation was to make us independent of imports for our nitrogen supply in case of war. An increase in the production of organic nitrogenous fertilizers would relieve the situation to a considerable degree. On the other hand, the high grades of organic nitrogenous fertilizers, cotton seed meal, gluten meal and dried blood, are finding more profitable use as cattle feed and are disappearing from the fertilizer market.

The nitrogen loss by the wasteful disposal of sludge from sewage purification processes is one of the greatest economic wastes in the civilized world today. The Metropolitan Sewerage Commission[25] of New York City, in 1910, estimated the dry suspended matter in the New York City sewage at 45 tons annually per 1,000 inhabitants. When it is considered that sewage sludge contains from 1.5 to 3 per cent nitrogen, 0.5 to 1.8 per cent phosphorus as P_2O_5 and 6 to 10 per cent fats, it is easy to see the economic waste in not recovering the vast supply of nitrogen and phosphorus which might be made available for use as fertilizer, and of fat which is so valuable at the present time. The chief

difficulty in the disposal of sludge from septic and Imhoff tanks as a fertilizer, is its high grease and water content.

A new method of sewage disposal[2] popularly known as "the activated sludge method" yields a sludge relatively high in nitrogen and phosphorus, and low in grease, but the moisture is very high and is held with great tenacity and methods of drying are still a serious problem in the preparation of the sludge for the fertilizer market. It has been shown[6] that the nitrogen is in a form available for plant food, and further studies, to be reported in this thesis, confirm and attempt to explain our first results.

Vast amounts of nitrogen are thrown away in the disposal of the sewage sludge from septic and sedimentation tanks, but a still larger amount is lost through anaerobic decomposition in these processes.[15] Nitrogen, ammonia and volatile amines are liberated by reduction and hydrolysis of the more complex solid organic nitrogenous substances. Furthermore, the colloidal material remaining in suspension after sedimentation is highly nitrogenous, and is wasted unless subsequently caught in sprinkling filters, where the oxidizing reactions prevent putrefaction. Sprinkling filter sludge contains about two or three times as much nitrogen as ordinary septic sludge (3 to 4 per cent), and recently engineers[34] have recommended supplementing sprinkling filters with humus tanks to recover this sludge.

The Miles acid method of sewage treatment[41] has recently been discussed in technical magazines because large quantities of grease may be extracted from the sludge, leaving a residue high in nitrogen which should be available as a fertilizer base. The nitrogen content of this sludge obtained by Dorr[41] working with a strong Boston sewage is 3.3 to 3.6 per cent, figured on a dry basis. In this process the sewage is nearly sterilized, and no loss of nitrogen by putrefaction and reduction should take place. This is borne out by the high nitrogen content of the sludge. On the other hand, much nitrogen in the colloidal form is not thrown down by this method of treatment.

In the aeration of sewage in the presence of activated sludge, a sludge is formed which is still higher in nitrogen content than those mentioned above. This is probably due to the more complete precipitation of colloidal matter in the sewage, and the limited action of aerobic bacteria upon the nitrogenous matter. Some have attributed the high nitrogen content to the destruction of carbonaceous matter by aerobic decomposition.[32]

In Table 1 is found a summary of chemical analyses of various types of sludges showing the variation in their nitrogen content.

TABLE 1—NITROGEN CONTENT OF VARIOUS SLUDGES.

	Per cent organic matter.	Per cent total N.	Per cent P_2O_5.
Septic tanks	29–54	1.1–2.9	----------
Emscher tank	38	1.2	----------
Filters—intermittent	----------	1.45	0.78
Chemical precipitation	----------	1.28	0.98
Grossmann's process	35	1.5	5.5
Globe fertilizer	43	1.65	1.71
Miles acid	----------	2.7 to 3*	----------
Milwaukee activated sludge	----------	4.5†	1.75
Urbana activated sludge	75	6.3	1.44
Urbana activated sludge	----------	4.59	----------
Chicago Packing House waste	----------	4.59	2.

* Calculated from NH_3 to N_2.
† Calculated as N_2.

HISTORICAL DEVELOPMENT OF SEWAGE SLUDGE AS A FERTILIZER.

The conservation of fecal material for fertilizing purposes was practiced years ago in China,[12] where the need was most pressing for nitrogen and phosphorus to fertilize the vast expanse of land. This most primitive and effective method of disposal has long been followed on the continent of Europe, and until quite recently has been employed on a large scale in Baltimore, Md. In 1909[1] Dr. J. M. Bosley, Commissioner of Health, reports that 61,748 loads (200 barrels each) of night soil were removed from cesspools and towed by barges down the Patapsco River for about eight miles, where the contents were pumped into lagoons prepared by farmers. These lagoons were used merely for storage until the material was required; then it was bailed with long-handled dippers into tank carts and sprinkled over the fields. Farmers reported that the liquid portion appeared to be more immediately effective but the heavier portion produced a more lasting effect. With the introduction of sewers this method of disposal, of course, is rapidly being abandoned.

The utilization of the fertilizing constituents of a city's sewage was first officially recognized in England by the Royal Sewerage Commission appointed in 1857, whose report in 1865 recommended land treatment of sewage but expressed doubt as to the profits of sewage farming. However, irrigation of land with sewage was a very old practice when this report was made. A famous example had existed for two centuries in Edinburgh.[24] Since the middle of the sixteenth century the sewage of Bunzlau, a German town, had been used for irrigating cultivated ground, and the same method had been practiced in Ashburton and other Devonshire towns from the beginning of the eighteenth century. A committee appointed by the local governing board in 1875 reported "that town sewage can be best and most cheaply disposed of and purified by the pro-

cess of land irrigation for agricultural purposes, where local conditions are favorable to its application." It was pointed out however that the fertilizing value to the farmer was greatly reduced because the sewage must be disposed of daily throughout the year. Sewage farms in England have not been successful from a financial point of view, and have gradually been replaced by chemical and biological treatment.

The largest and best managed sewage farms are those of Berlin and Paris. Data on the cost and return from the Berlin farms are given in the 1914 report of the Metropolitan Sewage Commission of New York. The expense for the year ending March 31, 1910, was:

Maintenance	$1,300,385.24
Interest and loans	741,817.62
Total	$2,042,202.86
Receipts for year	$1,240,772.58
Estimated increase of live stock and other property	122,593.50
Total	$1,363,366.08

This shows a total loss for the year of $678,836.78.

Calmette according to Clark[8] after studying the Paris farms concluded that they will be abandoned, and Dunbar believes that at Berlin artificial filters will some day take the place of the vast sewage farms now in use. In the United States sewage farming has not been considered favorably by our large cities but there are a number of smaller places where it is practiced with varying success.

Literature is full of reports on the availability of the nitrogen and phosphorus in sludge. The conclusion in the 5th Report (1908) of the British Royal Commission on Sewage Disposal was that, while sludge undoubtedly has a fertilizing value, the value is small compared with the gross mass of the sludge, and consequently depends on the cost of carriage. In Appendix 4 of this report Dr. J. A. Voelcker concluded that sludge on grass land had doubtful fertilizing value and that lime was needed to increase the availability of the nitrogen. T. H. Middleton, in the same appendix also reports that the sludge has no manurial value on grass land. More promising results were reported later by Dr. Voelcker after two years work with grain fields. Sludge applied at the rate of 40 pounds of nitrogen per acre increased the wheat crop 10 to 12 per cent in both straw and grain. Later experiments reported in the appendix of the Commission's Monthly Report showed that neither plain nor degreased sludge had any effect on the crops. But it is interesting

to note that in pot cultures untreated and degreased sludge increased the yield of wheat 25 and 14 per cent respectively, the degreased sludge proving inferior. In most of the reports of the Commission it is seen that sludge is of little value, and the unavailability of its fertilizing constituents is attributed to the large per cent of grease present, which forms an impervious layer or covering and hinders biological decomposition.

Naylor[26] attributed unavailability to grease and believes that it must be removed before sewage sludge will be accepted as a manure worthy of consideration by agriculturists. H. M. Wilson[42] of the West Riding Rivers Board states that these experiments and conclusions are opposed to practical experience, in view of the fact that farmers and market gardeners near the large cities continue to use sludge and can show that it has a marked effect on their crops, whether of hay, grain, roots, rhubarb or vegetables. He also claims that the agriculturists of France, Argentine and the United States make large purchases regardless of the cost of shipping. To quote Wilson, "If sludge is so valuable in these countries it should be well worth trying at home where transportation costs are much lower." This report mentions several municipalities in England with unusually rich sewage, which are able to sell part, if not all, of their sludge, and a few which add lime or other constituents to the sludge to make it marketable. At the Dalmarnock works in Glasgow, a chemical precipitation plant, the sludge is dried, ground, and sold under the name of Globe Fertilizer at $2.40 a ton. The Royal Commission on Sewage Disposal in its Fifth Report, page 162, comments thus on the success of the plant: "It will be seen, therefore, that at Dalmarnock by careful organization and advertising, practically the whole of the sludge is disposed of by sale, either as pressed cake or as Globe Fertilizer, the cost of the sewage treatment being in this way materially reduced. The saving effected is about 4 shillings per million gallons (Imperial). During the 3 years ending March 31, 1907, the whole of the pressed cake was sold to the farmers. The demand is still increasing and the connection is now good enough to obviate the necessity for advertising. Mr. Melvin states that it is much easier to sell sludge as pressed cake than as Globe Fertilizer, partly because less work in entailed in the former case ..... For this reason Globe Fertilizer is now made during spring and autumn only and the quantity is diminished year by year."

At Oldham, England, the Grosmann[10] process is used. This consists in drying, powdering, distilling with acidulated super-heated steam, and again drying. This plant began operation in 1912 and proved so satisfactory that in 1914 the Local Government Board sanctioned an

additional loan to complete parts of the plant which were not provided in the initial works.

At Bedford, England, the sewage contains a large quantity of grease from woolen mills. This grease is precipitated in the sludge by means of sulphuric acid and the sludge is heated to 100° with more acid and pressed to extract the grease. Part of the pressed sludge is used as fuel, part is sold to farmers for 84 cents a ton at the works, and part is dried and powdered, selling at $1.32 to $1.60 a ton (pre-war prices).

Sludge from the Travis tanks at Norwich[24] is treated by the Eckenburg wet carbonization process in which some of the grease is carried away by the hot gases and steam generated from the wet sludge. The sludge after this treatment drains easily and is filter-pressed and dried in rotary driers to 10 per cent moisture. The cake is extracted with benzine and dried. It finds a market at about $12 per ton of 2,240 pounds. The grease recovered is sold at $35 to $50 per long ton (pre-war prices).

Watson[40] mentions with approval the process at Dublin, Ireland. Here the sludge is incubated with 5 per cent brewer's yeast for 24 hours at 90°F. and drained, reducing the moisture to 82 per cent. Phosphates and potash are then added and the mixture dried and powdered.

H. W. Clark[8] from his study of the literature and his own work concludes: (1) sewage farming is rapidly being abandoned in favor of modern methods of purification; (2) in order to reclaim the valuable material from sludge it must be dried, degreased, and powdered. Any known method of grease extraction is costly. The sludge has a value, however, and as the processes of treatment are improved, sewage sludge will become of greater agricultural value than at present.

An important point is emphasized by H. Bach and L. C. Frank,[3] which may explain the reports of the poor fertilizing value of sludge. They show that in fresh sludge the grease and fibrous material cause the earth to become impervious to rain so that the sludge probably does more harm than good, while decomposed sludge contains less fat and is not so fibrous. Moreover, in the latter the fat, finely dvided and uniformly distributed through the mass of porous sludge, is not an appreciable hindrance to percolation; hence when such sludge is used as a fertilizer the prorosity of the ground is not affected. That the physical condition of a sludge is an essential factor in its usefulness as a fertilizer has been commonly overlooked.

Lipman and Burgess,[20] after extensive work on organic nitrogenous fertilizers, have introduced a biological method for the determination of availability of organic nitrogen. This method depends on the power of a soil to decompose and nitrify the organic material, the availability

being expressed in the percentage of total nitrogen that is converted into nitrate. Table 2 shows a comparison of availability of nitrogen in sludges and other standard organic nitrogenous fertilizers as determined by Lipman and Burgess in three typical California soils.

They have shown from the standpoint of nitrifiiability that (1) the nitrogen in mine sewage sludges and low grade tankage is superior to that of dried blood, high grade tankage, fish guano, cotton seed meal and goat manure; (2) soils differ greatly in their power to nitrify dried blood, high grade tankage, fish guano, cotton seed meal and goat manure; (3) nitrification of sludge and low grade tankage was more uniform. They conclude that if nitrifiability is a guide, sludge nitrogen is of equal value with low grade tankage, and of greater value than any other material named.

Bartow and Hatfield, in "The Value of Activated Sludge as a Fertilizer"[5] studied the physical, chemical and fertilizing properties of activated sludge. Their conclusions were that: (1) activated sludge has a chemical composition which indicates that it is far superior to ordinary sewage sludges as fertilizers; (2) the nitrogen is readily available, in fact more so than in other organic nitrogen fertilizers; (3) the physical character of the sludge makes it easy to handle: (4) the grease content of eleven per cent does not inhibit the fertilizing value, probably because it is finely divided and in no way clogs the soil; (5) the dried sludge contains only spore-forming bacteria; and (6) the nitrogen and phosphorus content calculated at prices in 1916 should give it a value of $34 per ton, while the cost of drying and preparing for market would be much less.

The most recent development in obtaining fertilizer and grease from sewage appeared in a proposal to the United States Government by experts from the Massachusett Institute of Technology, January 26, 1918.[29] The data presented showed that by the Miles acid process, there could be recovered annually from the sewage of 97 American cities, each with a population above 50,000, the following ingredients:

Fertilizer	97,393,680 tons
Ammonia	4,869,684 tons
Grease	25,780,680 tons
Glycerine	1,289,034 tons

The judgment was that an experimental plant should be tested at one of the army cantonments. On the other hand, the Massachusetts Joint Investigation Board,[23] appointed by the Massachusetts Legislature, 1917, reported January 1, 1918, against applying the Miles acid process to Boston sewage.

L. Pearse[80] shows that the activated sludge process is the best method for treating packinghouse waste in Chicago, that the sludge shows a high nitrogen content and that it should have considerable fertilizer value. The estimated value of recoveries from a plant treating 50,000,000 gallons per day amounts to $150,000 to $300,000 per annum calculated at normal prices.

THEORETICAL DISCUSSION OF ORGANIC NITROGENOUS MATERIAL AND ITS DECOMPOSITION PRODUCTS AS A FERTILIZER.

A brief history of the development of ideas on soil fertility serves best to show the inadequacy of some modern methods of soil and fertilizer analysis.

In the seventeenth century Van Helmont,[12] a Flemish alchemist, proposed the theory that water was the plant food, but later Bradley proved that water could be evaporated and plants could not. In the eighteenth century Jethro Tull taught that the soil particles were the food of plants, and that, if the soil were made sufficiently fine by cultivation, the plants could then absorb these fine particles. During the following century the humus theory advanced by Thaer gained some recognition. The humus of the soil was held to be the source of carbon and carbonaceous matter for the plant. In 1804 Dr. Saussure proved the fixation of carbon, hydrogen, and oxygen by photosynthesis, and the absolute necessity of mineral plant food. About this time Lawes and Gilbert proved that the soil must furnish the nitrogen for most plants and Hellriegel (1868) discovered the fixation of nitrogen by the root-tubercle bacteria of legumes.

At present it is generally considered that the ten essential elements of plant food may be grouped as follows: carbon, hydrogen and oxygen obtained chiefly from air and water; phosphorus, potassium and nitrogen, sometimes deficient in soils; and sulfur, calcium, iron and magnesium required in small amounts and not likely to be deficient in soils.

Of these we are intedested in the nitrogen, phosphorus, and organic matter, since they are constituents which would make activated sludge valuable as a fertilizer. These elements are bound in organic combination, and their availability to the plant will depend on the rate of decomposition of this matter in the soil. Agronomists classify organic matter in the soil in two classes, the active organic material which is easily decomposed and incorporated in the soil, and the inactive which resists decomposition, remaining for a number of years in the soil. No distinct line, however, can be drawn between these classes, the fertilizer practice has given us numerous availability determinations for elements, which arbitrarily set the line. These determinations are made on the theory

that a definite chemical reaction takes place in the soil which makes the easily hydrolyzable forms available for the plant. The available nitrogen is determined by distilling the ammonia from the fertilizer after digesting with a certain concentration of permanganate, in alkaline or neutral solution depending on the preference of the analyst and the statutes of the state in which he is working. The availability of phosphorus is determined by extraction of the phosphate with various concentrations of acids. Of course these procedures do not even approximate the conditions in the soil and are used because we have had no better methods at our disposal.

Plant food is made available by chemical and biological processes, of which ammonification and nitrification are among the best known. For the exact information we now have regarding these processes, we are indebted to Pasteur, Schlösing, and Müntz of France, Winogradsky of Russia and Warington of England. They have shown that complex organic nitrogen is first ammonified by certain classes of bacteria; then another class converts this ammonia into nitrite, and a third class transforms the nitrite into nitrate. It has recently been shown by Hopkins and Whiting[14] that these reactions increase the availability of insoluble phosphate. The nitrous and nitric acids as well as the organic acids formed in the hydrolytic and ammonification processes convert insoluble phosphates into soluble acid phosphates.

Because the availability of the nitrogen in soils is dependent on biochemical action and the well-known arbitrary chemical methods have no relation to the actual condition of availability of nitrogenous material in soils, bio-chemical methods of analysis have been introduced. In 1910 J. C. Lipman[21] proposed incubating soils with organic nitrogenous fertilizers and determining the ammonia and nitrate formed after a certain period of incubation. C. B. Lipman[19] points out that in some soils there is no connection between their ammonifying and nitrifying powers. He recommends incubation of one gram of fertilizer with 100 grams of soil at optimum moisture content for 30 days at 28° to 30°C., then nitrates are determined and the availability of the nitrogen expressed in the percentage of total nitrogen that is converted into nitrate. He has proved his point by comparison with pot culture experiments and recommends this method as the best one so far proposed, tho he says that the only absolute method is that of actual crop production.

A compilation of the data given by Lipman and Burgess[20] in their article on "The Utilization of the Nitrogen and Organic Matter in Septic and Imhoff Tank Sludges" and the analyses made for us by Professor Lipman using activated sludge are given in Table 2:

TABLE 2—PARTIAL COMPOSITION OF AIR-DRIED SLUDGES AND THEIR AVAILABLE NITROGEN COMPARED WITH THAT OF COMMERCIAL ORGANIC FERTILIZERS.

	Volatile matter, per cent.	Ash, per cent.	Total N., per cent.	Nitrate N., per cent.	Phosphoric acid, per cent.	Percentage of N. in sludges and fertilizers available when inoculated with—		
						Davis soil.	Oakley soil.	Anaheim soil.
Sludge—								
Orange Imhoff tank (city)	49.68	50.32	2.66	0.012	1.11	32.50	32.30	27.20
Fullerton Imhoff tank	25.31	74.69	1.23	0.045	0.86	43.90	43.50	40.60
Anaheim Imhoff tank	33.09	76.91	1.54	0.115	0.99	32.40	36.00	40.20
Lindsay septic tank	42.92	57.08	1.83	0.090	0.89	25.70	18.00	18.80
Pasadena Imhoff tank	29.34	70.76	1.68	0.135	1.46	38.00	28.20	35.70
Orange Imhoff tank (country)	38.41	61.59	2.38	0.060	0.77	25.70	21.40	15.70
Worcester exp. Imhoff tank	43.86	56.14	2.10	0.010	1.82	26.90	12.40	34.50
Cleveland exp. Imhoff tank	36.37	63.63	1.44	0.000	1.28	32.90	8.30	44.10
Chicago Stock Yards exp. Imhoff tank	50.46	49.54	1.73	0.400	1.46	24.50	9.80	10.10
Activated sludge	75.00	25.00	6.2	0.000	2.9	15.8	2.9	6.5
Fertilizers—								
Dried blood	------	------	------	------	------	12.79	0.00	4.05
High-grade tankage	------	------	------	------	------	16.21	0.00	3.95
Low-grade tankage	------	------	------	------	------	27.39	22.70	43.89
Fish guano	------	------	------	------	------	15.11	trace	4.65
Cottonseed meal	------	------	------	------	------	14.18	2.00	21.45
Goat manure	------	------	------	------	------	4.89	3.50	10.39

These results show that the nitrogen in activated sludge is more easily nitrified, under the conditions of these experiments, than that in dried blood, high-grade tankage, fish guano, cotton-seed meal and goat manure; but that it is not so available as that of septic and Imhoff tank sludges. The experiments with wheat in pot cultures using activated sludge, dried blood and other ammoniates, reported by us in 1916[5] and later in this thesis, confirm the above comparison between dried blood and activated sludge. On the other hand we have obtained much better results with activated sludge than with Imhoff and septic tank sludges (See Table 7.) Here this method seems to fail, as all arbitrary methods have done. A possible explanation for this lies in the fact that equal amounts of fertilizer are applied regardless of the nitrogen content; in the case of Imhoff and septic tank sludges 14 to 26 milligrams of nitrogen were applied while in the case of activated sludge 62 milligrams were added. A glance at the data[20] shows that in the three soils used, the nitrification capacity was from 1 to 9 milligrams of nitrate nitrogen. If it is true that a soil has a maximum capacity to produce nitrates, the percentage of the total nitrogen changed to nitrates will be an inverse function of the amount applied. On this basis the results on dried blood are unfair, since approximately 140 milligrams of nitrogen or 5 to 10 times as much nitrogen as that applied in sewage sludge was added in the form of dried blood. In comparison with activated

sludge, only twice as much nitrogen was applied in the form of dried blood nitrogen.

The terms ammonification and nitrification sound quite simple and might lead one astray. On second thought we realize that the organic matter in soils and organic nitrogenous fertilizers is in very complex form, and that its ammonification and subsequent nitrification must be preceded by reduction, oxidation, hydrolysis, and many other cleavages, each brought about by a specific group of organisms. The organic material containing nitrogen must be largely albuminoids and complex proteins (nucleoproteins, phospho-proteins and glyco-protein, and their various hydrolytic products.)

Schreiner and his associates[35] have isolated from soils the following nitrogenous hydrolysis products of proteins and other organic matter: (1) histidine; (2) arginine; (3) nucleic acid; (4) hypoxanthine; (5) xanthine; (6) guanine; (7) adenine; (8) asparagine; (9) creatinine; (10) creatin; (11) choline; (12) leucine; (13) allantoin, and (14) picoline carboxylic acid. Many other compounds in the soil have been isolated and studied but as they are non-nitrogenous we will not consider them. Schreiner and Skinner[36] have shown that, in water culture, wheat plants will directly absorb these compounds (except picoline carboxylic acid) and use them as food for growth without their first being changed into ammonia or nitrates. In the presence of nitrates the plants used not only the nitrate in preference but also some of the more complex compounds directly, and the plants grown in solutions containing both organic nitrogen and nitrate were better developed than those grown with nitrate alone. Some of Schreiner and Skinner's[36] data is compiled in Table 3 to show these results:

TABLE 3—PERCENTAGE INCREASE IN WHEAT CROP IN SOLUTION CULTURE WITH ADDED ORGANIC COMPOUNDS FOUND IN SOILS WITH AND WITHOUT NITRATE.

Organic nitrogen.	p. p. m. application.	No nitrate present.	With 8 p. p. m. NH_3 as nitrate.	With 16 p. p. m. NH_3 as NO_3.
Histidine	50	30	14	5
Arginine	50	33	0	0
Nucleic acid	100	74	23	12
Hypoxanthine	100	41	14	3
Xanthine	25	21	----------	----------
Guanine	40	5	----------	----------
Adenine	----------	----------	----------	----------
Asparagin	50	47	14	7
Creatinine	50	36	17	8
Creatine	50	44	11	3
Choline	Less	100 no effect	greater tha	n 100 toxic
Leucine	50	11	3	0
Allantoin	?	23	0	0

Results published in the bulletin show that even where nitrate is present, the above organic nitrogen compounds were absorbed directly by the plant and a decrease in the nitrate consumption took place, even though an increase in growth was shown. "The knowledge that beneficial organic compounds exist in the soil and play so prominent a part in the life processes of growing plants is of fundamental significance in soil fertility and gives a breadth of view to the subject, which, in its horizon, can not be compared with the restricted vision imposed by the purely mineral consideration."

Of course conclusions drawn from solution cultures can not necessarily be applied to soil conditions because the soil is very complex and the bacterial flora is equally so. The chance of absorption by colloidal substances, and of bacterial decomposition in the soil is so great that we must guard against false deductions. Nevertheless it is significant that the plant will use these products if they are available.

R. C. Lathrop[18] discusses in his paper, "The Organic Nitrogen Compounds of Soils and Fertilizers," the nature of organic compounds in the soil. It is shown that the compounds isolated by Schreiner and also by himself are obtained by the hydrolysis of the complex protein-like compounds occurring in soils and organic fertilizers. He shows that the rates of ammonification and nitrification are important, and that they are dependent on the rate and type of hydrolysis and hydrolic products. To quote Lathrop, "It is obvious that a knowledge of the normal decay of protein material in soils will materially aid in solving the availability problem." In his study of the hydrolysis of organic matter in the soil and of dried blood in the same soil, Lathrop used the Van Slyke method of nitrogen partition. He has shown that dried blood is rapidly hydrolyzed in the soil, as indicated by the decrease in hydrolyzable nitrogen found after, 18, 24, 86, 148 and 240 days. It is emphasized that the intermediate products are beneficial to plant growth and that so far we have no chemical method of determining availability.

The decomposition of such a complex protein as nucleo-protein may be taken as an example to show the intermediate and final products, so far as we know the reactions in biochemistry. The complete enzymatic decomposition of nucleoprotein into its intermediate and final products in so far as animal metabolism is concerned, may be found in Jones Monograph.[16] The final products are amino acids, uric acid or allatoin, purine and pyrimidine bases, carbohydrate, and phosphoric acid. These may all undergo bacterial decomposition in the soil to form ammonia and carbon dioxide.

The ammonia thus formed may be converted into nitrite and thence into nitrate by nitrifying bacteria. The opportunity which a plant

would have to absorb any intermediate product would depend partly on the solubility of the substance and partly on the speed with which it is converted into a still simpler compound.

The above considerations may explain the results obtained in pot culture. The results obtained showed the superiority of activated sludge to dried blood, glutonmeal, sodium nitrate and ammonium sulfate, when used on wheat in pot culture. In all the experiments dried blood has been used for comparison because it is generally considered the best organic ammoniate. Several factors probably affect these results. (1) The large amount and the type of organic matter applied in activated sludge may favor the growth of a better flora of microorganisms to ammonify and nitrify the nitrogenous matter; (2) the organic matter itself may be more easily hydrolyzed; (3) it has been suggested that in dried blood the organic matter is too easily ammonified and that the high concentration of ammonia inhibits the growth of the nitrifying bacteria; (4) if this be true the superiority of activated sludge may be due to slow hydrolysis, which gives the nitrifying bacteria time to convert the ammonia into nitrates as rapidly as it is formed; (5) possibly too high concentrations of dried blood were used; and (6) the nature of the organic matter and its intermediate hydrolytic products may play a prominent part in inhibiting or stimulating the growth of the plant. These explanations have all been considered and an attempt made through laboratory analysis and pot culture studies to verify them.

These experiments with one exception have all been made with dried sludge. The sludge as obtained from the process contains 98 to 99 per cent moisture. The reduction of the moisture content is now one of the most pertinent problems in the practical operation of the activated sludge process of treatment. The colloidal, gelatinous nature of the wet sludge makes the problem of dewatering more complex, and if not dewatered while fresh the sludge soon putrefies and becomes still more difficult to handle. Also if the sludge is to be marketed it must be converted into an attractive, saleable form and must contain less than 10 per cent moisture. The various precipitants and methods of filtering and drying tried in this laboratory are reported below.

EXPERIMENTAL PART.

The Application of Fertilizers to Plants in Pot Culture and Garden.

The results on the first series of pot cultures were reported in 1915.[5] Thirty wheat seeds were planted, two in each of fifteen holes in white sand to which the necessary plant foods were added as shown in Table 4. After germination the young wheat plants were weeded out, leaving the best fifteen plants in each pot.

TABLE 4—POT CULTURES, USING WHEAT, SERIES 1, 1915.

Plant food in grams.							Yield.				
Pot No.	Dolomite.	Bone meal.	Potassium sulfate.	Activated sludge.	Extracted activated sludge.	Dried blood.	Grams seeds.	Bu. per acre (calculated).	Av. stalk length (in.).	Straw grams.	Tons per acre (calculated).
1	60	6	3	0	0	0.0	2.38	6.2	19.4	2.25	0.18
2	60	6	3	0	0	8.61	5.29	13.6	23.0	8.25	0.68
3	60	6	3	20	0	0.0	13.748	35.9	35.4	26.75	2.23
4	60	6	3	0	20	0.0	14.504	37.7	36.1	26.21	2.18

19,820 grams of white sand were used in each pot.

TABLE 5—RETURNS FROM POT CULTURES, SERIES 2, 1915.

GROUP I.

No.	Fertilizer used.*	Application.		Nitrogen equivalent.		Grain.		Straw.		
		Grams.	Tons per acre.	Grams.	Lbs. per acre.	Weight grams.	Bu. per acre.	Weight grams.	Tons per acre.	
1		0	0	0	0	2.7	7.0	1.9	.2	
2	Activated sludge	20	1.7	1.26	210	13.5	35.1	22.2	1.8	
3	Fat-free activated sludge	19.14	1.6	1.26	210	13.3	34.6	22.4	1.8	
4	$NaNO_2$	2½	.2	.41	68	.5	1.3	2.3	.19	Toxic**
5	$(NH_4)_2SO_4$	5.94	.5	1.26	210	1.8	4.7	5.0	.4	
6	Gluten meal	16.93	1.4	1.26	210	3.7	9.6	6.7	.6	
7	Dried blood	8.61	.7	1.26	210	3.6	9.4	5.4	.4	

GROUP II.

1		0	0	0	0	2.9	7.5	2.5	.2	
2	Activated sludge	30	2.5	1.89	315	13.0	33.8	19.6	1.6	
3	Fat-free activated sludge	28.72	2.4	1.89	315	14.2	36.9	25.1	2.0	
4	$NaNO_2$	2½	.2	.41	68	1.5	3.9	5.0	.4	Toxic**
5	$(NH_4)_2SO_4$	8.91	.7	1.89	315	2.85	7.4	7.9	.7	
6	Gluten meal	25.39	2.1	1.89	315	8.6	22.1	13.7	1.1	
7	Dried blood	12.91	1.1	1.89	315	3.9	10.1	7.7	.6	

* To each pot of white sand, plant food was added as follows: 60 grams of dolomite (5 tons per acre), 6 grams of bone meal (½ ton per acre) and 3 grams of potassium sulfate (¼ ton per acre).

** The sodium nitrate seemed to produce a toxic effect in this concentration, so no more was added as had been anticipated.

Activated sludge applied on an equivalent nitrogen basis produced over twice the grain and straw produced by dried blood. Before the final results on this series of pot cultures were obtained, a second series[5] was started in order to check the first results. The second series was made more extensive, consisting of fourteen pots divided into two groups of seven each, and compared the availability of the nitrogen in activated sludge, dried blood, gluten meal, sodium nitrate and ammonium sulfate. The same amounts of sand and plant foods, except nitrogen, were used as in the first series. Each group contained a check pot to which no

nitrogen was added. To the other six pots of the first group nitrogen equivalent to twenty grams of activated sludge was added as sludge and as the other fertilizing materials indicated above. In the second group nitrogen equivalent to thirty grams of activated sludge was used to determine if a large application would produce better results. Wheat was planted in this series in the same maner as in Series 1. A picture of this series, when 5 weeks old, was published in 1915, but final results on crop recoveries were not ready at the time of publication. Table 5 contains complete data on plant food application and crop production.

The data show again that activated sludge gave better results than dried blood in production of both grain and straw. In the low application it gave much better results than gluten meal, while in the higher application this difference was not so marked. The results obtained with sodium nitrate and ammonium sulfate were unimportant. The concentrations used should not have been toxic; in fact only part of the nitrate was added at first in order to avoid toxicity. Again the results indicated beyond any reasonable doubt that the nitrogen in activated sludge is very readily available to the plant. The difference in high and low application was minimal. With gluten meal the higher application produced a substantial increase in yield over that from the lower application. In the case of dried blood the increased application showed no material improvement. The slight results obtained with the mineral nitrogen fertilizer may have been partially due to the lack of organic matter present to furnish food for the proper microscopical flora. It seems significant at least that the organic nitrogen cultures all surpassed those fertilizers with inorganic nitrogen alone. As these results with sodium nitrate are not in accord with general agricultural practice the subject should be investigated. Further results on nitrate cultures are reported in Series 6 (1918), Table 17.

In both Series 1 and 2 the degreased sludge showed no material increase in yield of crops over that of the original sludge. The results obtained in a garden experiment with lettuce and radishes, using both plain and degreased activated sludge, are given in Table 6 and show a slight increase in favor of the degreased sludge.[5]

TABLE 6—COMPARISON OF THE LETTUCE AND RADISHES FROM UNFERTILIZED AND FERTILIZED PLOTS.

Plot.	Treatment.	Weight.	Per cent increase.	Weight.	Per cent. increase.
1	None	4.5g.		23.4g.	
2	Sludge	6.3g.	40	63.0g.	169
3	Extracted sludge	6.8g.	51	68.0g.	190

The extraction of grease in preparing activated sludge for a fertilizer appears to be unnecessary. The fat present in activated sludge is so finely divided and evenly distributed thru the mass that it causes no greasiness and would not hinder the decomposition of sludge in the soil. On the other hand the present demand for fats may make it expedient and profitable to recover them. Further results on acidified sludge from which the fat has been extracted are reported in Series 6 (1918), Table 17.

It is difficult to reconcile the discordant estimates of the value of sewage sludge as a fertilizer. The fact that a few places, taking the trouble to properly dry and powder sludge have successfully sold it makes it appear that possibly some results in pot cultures with different sludges on different soils would be of value. In the spring of 1916 Series 3 was started. Four sludges were used in this experiment.

1. Imhoff tank sludge from the city of Baltimore, which had been dried on sand beds, and contained considerable sand which lowered its nitrogen value as a fertilizer was further dried on a steam bath in this laboratory and ground in an ordinary coffee mill. It was light gray in color and had little or no odor.

2. Septic tank sludge was collected from the Urbana septic tank, January 29, 1916. As most of the sludge in the tank had risen to the surface it was necessary to collect both the surface sludge and that from the bottom. An average sample was obtained and dried on a steam bath. The color of this sludge was a very light gray and the odor was quite foul.

3. Activated sludge was collected after 30 minutes' settling, from the "fill and draw" activated sludge tanks described by Bartow and Mohlman.[6] As much water as possible was separated by sedimentation and the resulting sludge was dried as described above. When dried, the sludge was brownish-black and had a characteristic fertilizer odor.

4. A sample of activated sludge from Milwaukee did not arrive until after the experiments were started. This sludge was filter-pressed in Milwaukee, and dried on a steam bath after its arrival. It had the characteristic fertilizer odor and brownish-black color.

The dried blood used in this series was from the same sample as that used in Series 1 and 2. The analyses for nitrogen in these fertilizers are:

Inhoff sludge	0.9 per cent
Septic tank sludge	1.49 per cent
Activated sludge (Urbana)	4.47 per cent
Milwaukee activated sludge	4.63 per cent

The three types of soil used in this series were obtained from the Agronomy Department of the College of Agriculture of the University of Illinois and represent three characteristic types of Illinois soil. They are known as yellow silt loam on tight clay, white silt loam on tight clay, and brown silt loam on tight clay. The analyses of these soils (Table 7) give an idea of the elements which they lack.

Average pounds per acre in 2,000,000 pounds of surface soil.

TABLE 7—ANALYSIS OF THREE TYPES OF ILLINOIS SOILS.[13]

Soil type.	Total organic carbon.	Total nitrogen.	Total phosphorus.	Total potassium.	Soil acidity.
Yellow silt loam	22,110	2,068	696	36,024	940
White silt loam	14,860	1,360	660	30,120	1,400
Brown silt loam		7,000	1,200	35,000	0

On February 7, 1916, three groups of six 4-gallon pots were filled, one group with each soil, and to the yellow and white silt loam 60 grams of dolomitic limestone, equivalent to 5 tons per acre, were added to neutralize the acidity of the soil. The fertilizers to be tested were added on an equivalent nitrogen basis and were plowed into the top six inches of soil. Thirty wheat seeds were planted in fifteen holes in each pot. The Milwaukee activated sludge was not plowed in and planted until March 20 and hence did not give results comparable with the rest of the series. The sludges were added in amounts equivalent to the nitrogen in 15 grams of dried blood, reserving one pot in each soil as a check pot. The actual quantities of sludges used are shown in Table 8. After complete germination some plants were removed, leaving the fifteen best plants in each pot. Activated sludge and dried blood slightly hindered germination in the brown silt loam, tho the plants which germinated were not hindered in growth. In those pots fertilized with septic tank sludge both germination and growth were retarded.

After four weeks the pots of yellow and white soils fertilized with activated sludge were by far the best, those fertilized with Imhoff sludge next, then those fertilized with dried blood and the check pot. Those fertilized with septic tank sludge were not so far along as the check pots. The pots containing the brown silt loam, a rich soil to begin with, were all good. Some trouble was experienced with a white mold which attacked the leaves of all the plants, but this was eventually overcome and did not seem to cause any permanent injury.

On April 10, when the plants were about two months old, pictures were taken of the white and yellow silt loam cultures. Figures 1 and 2 are pictures of yellow and white silt loam groups respectively. The

leaves of the rapidly growing plants are tender and do not hold themselves up as good, strong plants should. This is characteristic of plants that are overtreated with organic nitrogen fertilizer.

During May the plants fertilized with septic tank sludge began to surpass those fertilized with Imhoff sludge. The final results on the crop are given in Table 8.

Figure 2.—White Silt Loam Group: Plants About Two Months Old.

The data shows that, altho the plants fertilized by Imhoff sludge grew rapidly at first and gave an increase in straw, a decrease in grain production on the white and brown silt loam resulted. In the yellow silt loam a substantial increase in grain was obtained. Septic tank sludge caused a material increase in grain on the yellow and white silt

loams and a slight decrease on the brown soil. The production of straw was increased about 50 per cent on all three soils. The Urbana activated sludge increased the yield of grain far above that of the other fertilizers added, the same being true in production of straw. On the brown soil, which is a rich type and produced a good crop without fertilizer, both activated sludge and dried blood gave an increase in yield of wheat and straw. On the other soils dried blood fell far below the activated sludge in fertilizing value both in grain and straw production. With wheat planted a month and a half late the results with Milwaukee activated sludge, show an increase in straw, but only a slight increase in grain on the yellow soil and a decrease in grain on the other two soil types.

TABLE 8—DATA ON SERIES 3, 1916.

Fertilizer,	Fertilizer.		Bushels of wheat per acre.			Tons of straw per acre.		
	Grams.	Tons per acre.	Yellow soil.	White soil.	Brown soil.	Yellow soil.	White soil.	Brown soil.
Check	0	0	10.7	17.0	30.6	1.1	1.5	3.0
Imhoff	244	19.5	18.8	13.5	21.3	1.9	1.6	4.0
Septic tank sludge	147	11.8	23.8	22.3	27.3	2.7	2.5	4.6
Activated sludge	49	3.9	33.8	27.4	41.3	3.2	3.3	5.1
Dried blood	15	1.2	15.6	17.0	41.4	1.1	1.6	5.0
*Milwaukee activated sludge	47.25	3.8	14.5	16.8	27.3	1.9	1.6	3.0

* Results not comparable with others because of late planting.

Residual nitrogen in the soils was determined according to the regular method for total soil nitrogen and is expressed in Table 9 in pounds of nitrogen per acre.

TABLE 9—POUNDS PER ACRE OF RESIDUAL NITROGEN IN SOILS.

Fertilizer applied.	Soils.		
	Yellow.	White.	Brown.
Check	1,100	840	6,800
Imhoff	1,400	1,000	7,400
Septic tank	1,500	1,340	7,600
Urbana activated sludge	1,340	lost	7,400
Dried blood	1,160	890	7,600
Milwaukee activated sludge	1,260	1,400	7,400

These results compared with Table 7 show approximately the nitrogen recovered by the crops and lost to the air.

On May 22, 1916, a fourth series of eight pots was started, using a light gray silt loam from Lawrence County, Illinois. The pots were treated with an equivalent of 5 tons per acre of dolomitic limestone, and

were fertilized with various amounts of activated sludge equivalent to an application of from zero to 3 tons. On two pots, dried blood equivalent to 1.0 ton was used. One of the samples of dried blood was the same as that used in our earlier experiments, and the other was a new sample obtained from Swift and Company which contained 13.9 per cent nitrogen. Ten seeds of bantam sweet corn were planted, two each in five holes in each pot. After the plants were up the number in each pot was reduced to the five best plants. In this series the corn was not harvested, but the mature plants were cut at the surface of the ground and the dry weights taken. Data on the fertilizer application and crop yield are given in Table 10.

TABLE 10—DATA ON BANTAM SWEET CORN GROWN IN LIGHT GRAY SILT LOAM.

No.	Activated sludge.		New Swift dried blood.	Old dried blood.	Weight of crops.		Per cent increase over check.	Residual N. in pounds per acre in soil.
	Grams.	Tons per acre.			Grams.	Tons per acre.		
1	0	0	0	0	29	2.3	0	440
2	7.5	0.5	0	0	34	2.7	17.4	720
3	15.	1.0	0	0	39	3.1	34.8	800
4	22.5	1.5	0	0	57	4.5	95.6	880
5	30.0	2.0	0	0	41	3.3	41.3	lost
6	45.0	3.0	0	0	56	4.4	91.5	900
7	0	0	15 gr. (1 ton)	0	39	3.1	34.8	lost
8	0	0	0	15 gr. (1 ton)	33	2.6	13.0	1,200

This series shows that on this light gray silt loam 1.5 tons of activated sludge gave the best results, tho higher application did not prove toxic. The new dried blood showed better results than the old. One ton of the new dried blood gave the same results as one ton of activated sludge.

On July 9, 1916, a garden experiment was started near the Illinois State Water Survey sewage experiment station east of Urbana. The soil was a poor yellow clay which for many years had been unproductive. Four plots twenty-eight feet square were plowed and two of them fertilized with an equivalent of 1.22 tons of activated sludge per acre (102.5 pounds of nitrogen per acre), equivalent to about 0.4 ton of dried blood. The other two were not fertilized. Cucumbers and evergreen sweet corn were planted in the plots. On September 18, all cucumbers were harvested, and an increase of 71 per cent over the unfertilized plot was ascribed to activated sludge. The corn at this time was not ready to be harvested and was allowed to remain; later it was accidentally destroyed and results cannot be given. From all appearance the corn would have

shown an increase due to sludge equal at least to the cucumber increase just mentioned.

In the Series 5 in 1917, the plan was: first, by a study of the apparent toxic effect of dried blood to explain, if possible, the small results obtained with it in the earlier work; second, to study the effect, if any, of applying the different sludges as fertilizer to the same ground on the second year; third, in order to eliminate the question of the possible advantage of activated sludge over dried blood because of its phosphorus content, to add sufficient acid phosphate to one series so that phosphorus would not be the limiting element; and fourth, to see if toxic effects on wheat could be obtained by adding excessive amounts of activated sludge.

Figure 3.—This picture taken five weeks after germination shows the toxic effect of dried blood in sand as the amount applied increases. Pots 5 and 6 were not photographed since the plants were killed soon after germination.

First—The effect of increasing applications of dried blood was studied in sand culture containing all elements for plant food as in Series 1 and 2. The new sample of Swift's dried blood was then added in increasing amounts and wheat sown, with the results shown in Figure 3 and Table 11.

TABLE 11—WHEAT WITH INCREASING AMOUNTS OF DRIED BLOOD IN SAND.

No.	Dried blood.		Lbs. N_2. per acre.	Total crop.	Weight of grain.	Bushels per acre grain.	Total straw grams.	Tons straw per acre.
	Grams.	Tons.						
1	3.1	0.25	69.5	34.1	11.4	29.6	22.7	1.8
2	9.3	0.75	305.8	36.8	10.0	26.0	26.8	2.1
3	18.75	1.5	417.0	21.0	0.0*	0	21.0	1.6
4	37.5	3.0	838.0	16.4	0.0*	0	16.4	1.3
5	75.	6.0	1,670.0	0	0.0	0	0.0	0.0
6	125.	10.0	2,780.0	0	0.0	0	0.0	0.0

* Heads empty.

In sand, dried blood is toxic in applications above 0.75 of a ton, and that one-half a ton gave the largest yield of grain. In the pots containing the 1.5 and 3.0 tons treatment, plants grew but the heads contained no grain; in the still higher applications the blood was so toxic that the plants were killed before they completed germination. In soils this toxicity might not be shown at such low concentrations because the colloidal nature of the soil might adsorb and destroy more toxic substances than the sand. Nevertheless these results indicate that we may have used too much in some of our earlier work.

Second—The yellow silt loam pots of Series 3 were again fertilized with the same sludges and dried blood at about the same concentrations that were used the year before in order to determine if there would be any detrimental effect due to the accumulation of toxic substances. Wheat was planted as described above. Table 12 gives the data showing the amount of fertilizer applied, and the final crop harvested.

TABLE 12—DATA ON SECOND YEAR CROPS OF YELLOW SILT LOAM FERTILIZED WITH SLUDGE AND DRIED BLOOD.

No.	Fertilizers.	Application.		Total weight.		Bushels per acre of grain.	Weight of straw.	Tons per acre, straw.
		Grams.	Gr. N_2 applied.	Crop.	Grain.			
1	Check	0	0	10.6	4.0	10.4	6.6	0.5
2	Imhoff	120	2.12	12.8	4.6	12.0	8.2	0.6
3	Septic tank	70.6	2.12	31.8*	9.4*	22.4*	22.4	*1.8
4	Urbana activated sludge	42.4	2.12	31.7	6.4	16.5	25.3	2.0
5	Dried blood	15.0	2.12	9.1	2.5	6.5	6.6	0.5
6	Milwaukee activated sludge	52.3	2.12	16.8	5.5	14.3	11.3	0.9

* By mistake this pot was planted with bearded wheat and consequently is not comparable.

The sludges caused an increase in grain and straw, whereas dried blood was detrimental to grain production and had no effect on the straw. If these results are compared with those on the same soil in Series 3 (1916), it is seen that the increase due to fertilizing is considerably less in the second year. This is particularly noticeable with dried blood. We may expect crops to vary from year to year according to seasonal conditions, but in this case it seems probable that too much fertilizer had been added. The results, however, do not show that the extra application caused accumulation of substances toxic to the plants except in the case of dried blood.

Third—The white silt loam pots of Series 3 (1916) were fertilized on February 12, 1917, with the equivalent of one ton of acid phosphate per acre and nitrogen in the same forms and quantities as used the year

before. Wheat was planted as in previous experiments. The object of adding the acid phosphate, as stated above, was to eliminate the possible advantage activated sludge might possess by virtue of its phosphorus content. The data on this series are given in Table 13.

TABLE 13—DATA ON SECOND-YEAR CROP GROWN IN WHITE SILT LOAM TO WHICH ACID PHOSPHATE AND FERTILIZER HAVE BEEN ADDED.

Fertilizer.*	Application.		Total weight.		Bushels per acre.	Weight straw grams.	Straw tons per acre.
	Grams.	Gr. N_2.	Crop.	Grain.			
Check	0	0	30.4	9.8	25.5	20.6	1.6
Imhoff	12.0	2.12	19.8	4.0	10.4	15.8	1.3
Septic tank	70.6	2.12	34.6	7.7	20.0	26.9	2.1
Urbana activated sludge	42.4	2.12	30.2	12.6	32.8	17.6	1.4
Dried blood	15.	2.12	27.2	5.6	14.6	21.6	1.7
Milwaukee activated sludge	52.3	2.12	36.8	7.6	19.8	29.2	2.3

* To each pot an equivalent to one ton of acid phosphate was added.

Activated sludge was the only fertilizer that increased the yield of wheat over that on the check pot. It appears that phosphorus was the lacking element in this series, and that in the first experiments in this soil phosphorus, not nitrogen, was the limiting element. Also the toxic effect of a second application of Imhoff sludge, septic tank sludge, dried blood, and to a certain extent Milwaukee activated sludge, is plainly seen. Here again there appears to be an accumulation of toxic compounds from dried blood as well as from the anaerobic sludges, and no toxic effect from Urbana activated sludge.

TABLE 14—WHEAT GROWN ON ACID GRAY SILT LOAM FERTILIZED WITH ACTIVATED SLUDGE AND DRIED BLOOD (1917).

Application.					Total weight.			Bushels grain per acre.	Tons straw per acre.
Activated sludge.		Dried blood.		Lbs. N_2	Crop.	Seeds.	Straw.		
Grams.	Tons per acre.	Grams.	Tons per acre.						
6.3	0.5	----------	----------	50	34.4	13.1	21.3	34.1	1.7
12.5	1.	----------	----------	100	31.0	10.2	20.8	26.4	1.6
25.0	2.0	----------	----------	150	35.1	9.4	25.7	22.4	2.0
50.	4.0	----------	----------	200	40.8	8.9	31.9	23.1	2.5
100.	8.0	----------	----------	400	50.5	10.3	40.2	26.9	3.3
200.	20.	----------	----------	2,000	45.5	10.3	35.2	26.8	2.8
0	0	15	1.2	334	16.6	6.1	10.5	15.9	.8
0	0	15	1.2	334	14.1	4.4	9.7	11.5	.77

Fourth—The same soil used in Series 3 to study the effect of inincreasing amounts of activated sludge on corn was used in this group.

(See Table 10, p. 45). The purpose of this test was to determine again the effect on plants of increasing the amounts of activated sludge, and incidentally the cumulative effect of the repeated use of high amounts of activated sludge. The soils were plowed up and the second application mixed with the top 6 inches of the soil. Wheat was planted and the final results tabulated in Table 14.

The production of straw is increased by an application of activated sludge up to eight tons per acre, and the yield from 20 tons falls only a little below that from the 8-ton application. It is safe to say that even very high applications of activated sludge are not toxic so far as the production of straw is concerned. The yield of grain, however, is materially affected, for each successive increased application of activated sludge decreased the yield of grain. The best results were obtained with the application of one-half a ton; but it must be remembered that in the

Figure 4.—Effect on plants of increasing amounts of activated sludge.

previous year this pot had received no sludge and the high yield may have been due to this fact, since it has been shown that on the second application the yield of wheat grains is reduced. The other pots had received a somewhat similar application of sludge the year before, the rate of application being in the same order tho not the same quantities. It appears that 1.2 tons of dried blood was not equivalent in fertilizer value to one-half a ton of activated sludge; and that 1.2 tons of dried blood produced a greater toxic effected on wheat than did 20 tons of activated sludge. Figure 4 shows this series at the end of five weeks.

On May 16, 1917, two large garden experiments were started west of the activated sludge experimental plant which was east of Urbana. One of the garden plots was just west of the experimental plant. The soil of the plot was worn-out brown silt loam which had been used as a potato patch for some years. It had received no fertilizer in five or six years and had produced poor potatoes all of this time. Half of this plot

was fertilized by pumping wet activated sludge from the plant directly on the ground and plowing it in two or three days later. With this method of application the quantity of fertilizer applied cannot be given. The object was to try the application of wet sludge for fertilizing purposes, since in many small installations the expense would be materially reduced if the sludge could be disposed of without drying. This plot will hereafter be designated as the "wet sludge plot."

One-half of the second plot, to be known as the "dry sludge plot," was fertilized with a mixture of dried activated sludge from Urbana and Milwaukee. The other half of the plot was left unfertilized as a control. The soil of this plot was a poor yellow clay, the same type of soil used in the garden experiments reported above for the summer of 1916.

Radishes, lettuce, beets, onions and sweet corn were planted in both plots on May 20, 1917. Tomato plants were set out in the dry sludge plot on May 23 and 24 but due to the scarcity of these plants we were unable to set them out in the wet sludge plot until the last of June. Results on the tomato crop from this plot were not obtained before the early autumn frost. Data on these experiments are given in Table 15.

TABLE 15—EFFECTS OF DRY AND WET SLUDGES ON GARDEN PRODUCTS.

Crop.	Wet sludge plot per cent increase over check.	Dry sludge plot per cent increase over check.
Radishes	28	14
Lettuce	100	36.3
Beets	135	lost
Onions	10.0	6.4
Corn (green)	51.8	46.0
Corn (ripe)	23.0	6.7
Tomatoes	283.0	frost

While the comparison between the two plots is not entirely justified because sludges from different sources were applied and soils of different types were used, wet sludge was favorable to plant growth and gave much better results than the sludge used on the dry sludge plot, which was made up mostly of Milwaukee activated sludge. The results on the beet crop from the dried sludge plot were not obtained. Figures 5 and 6 show the quality of sweet corn produced on the fertilized and unfertilized plots. The ears from the fertilized plot were all good while most of those from the unfertilized plot were small and wormy.

A discussion of Series 6 and 7 planted in February, 1918, will follow the presentation of studies in the chemical constitution of the nitrogenous organic matter in activated sludge.

Figure 5.—Corn from fertilized plot.

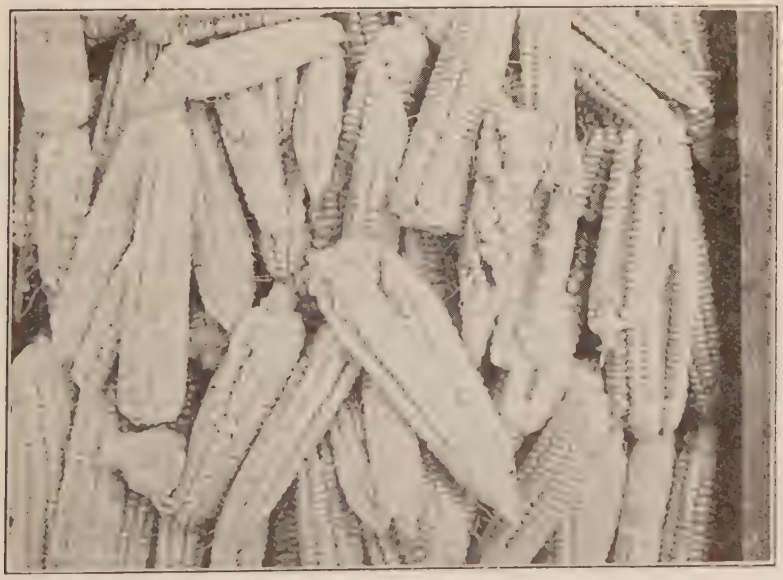

Figure 6.—Corn from unfertilized plot.

THE CHEMICAL CONSTITUTION OF THE ORGANIC NITROGENOUS COMPOUNDS IN ACTIVATED SLUDGE.

The question of the nitrogen chemistry of sludges involves the study of a mixture of very complex chemical compounds which have been subjected to a multitude of physical, chemical and bio-chemical influences which change both their physical and chemical nature. The first question is that of the original source of the materials in sludge, and then so far as possible, prediction of results of the many biological changes occurring under conditions which govern the formation of various sludges.

Sewage sludge is made up of substances which separate from sewage under certain conditions and therefore our first knowledge must be gained from a study of the chemical nature of sewage. It is estimated[24] from data on several sewages that the total solids in residential sewage are derived from the constituents as follows:

	Grams per capita per day.
Total solids in city water supply (soft)	12.7
Faeces	20.5
Urine	43.3
Toilet and newspapers (suspended)	20.0
Sinks, laundries, wash water	77.5
Total for residential sewage	174.0

The total solids of the city waters will vary considerably but do not enter into this discussion since they are soluble and do not form sludge. The feces, however, are largely insoluble and form a large part of the sludge. Solids classed as urine are soluble and may be eliminated from the discussion. Paper, tho it becomes very finely divided, must be classed as insoluble, sludge-forming material. Only a small part of the solids from sinks and laundries need be considered since they are largely soluble. The solids from street washing are of a gritty nature and could be removed in grit chambers. Therefore, it is plain that sewage sludge consists of fecal matter and paper in some stage of decomposition.

Fecal matter[11] is composed of the following substances: (1) food residues; (a) those portions of the food which have escaped absorption, (b) that part of the diet either not digested or incapable of absorption; (2) the remains of the intestinal and digestive secretions not destroyed or reabsorbed; (3) substances excreted into the intestinal tract, notably salts of calcium, iron, and other metals; (4) the bacterial flora of the intestinal tract; (5) cellular elements, to which may be added under pathological conditions, blood, pus, mucus, serum, and parasites; (6) abnormally; enteroliths, gallstones and pancreatic calculi.

Of these we are interested only in the nitrogen compounds, so a study of the various forms of protein and pigments present will throw considerable light on the question. These may be divided into the following groups: (1) simple proteins; albumin, globulin, casein and their decomposition products; (2) conjugated proteins; nucleo-proteins, nucleic acid, etc., glycoproteins (muscoids); (3) albuminoids; keratin, elastin, collagin; (4) pigments, etc.

Group 1 may be eliminated from the discussion since these proteins are found in the feces only under pathological conditions. The compounds in Group 2 are present in the bacterial bodies and cellular material in the feces. These form a large portion of the total fecal nitrogen. The albuminoids of Group 3 are the unabsorbed food residues mentioned above. The pigments of Group 4 are derived chiefly from the intestional secretions.

The data given below on the composition of feces was compiled from "Die Faeces des Menchen" by Schmidt and Strasburger,[39] "Faeces of Children and Adults" by Cammidge,[7] and "Die Analyse des Harns" by Neubauer.[28]

Per cent of dried feces.

Total nitrogen, 8 to 9, normal ammonia in feces, 0.151, ether soluble extract, 16 to 17, ash 13 to 14.

Per cent of total nitrogen.

Soluble nitrogen, 32.0, bacterial nitrogen, 30 to 50, purine nitrogen, 10.

MacNeal, Latzer, and Kerr[22] have shown that the total nitrogen in dried feces of men averaged 11.69 per cent, the per cent of dried bacteria in feces was 29.86 per cent and the per cent bacterial nitrogen of the total nitrogen was 46 to 48 per cent.

Now if 32 per cent of the nitrogen in feces is soluble, 47 per cent is bacterial nitrogen, and 10 per cent is purine nitrogen, there remains 11 per cent of nitrogen from insoluble food residues. The insoluble nitrogen, therefore, which separates from sewage in the form of sludge would consist of 47 parts of bacterial or nucleo-protein nitrogen, 11 parts of other organic nitrogenous matter, and that portion of the 10 parts of purine nitrogen which is insoluble or 69, 16 and 14 per cent respectively. Of this 16 per cent of organic nitrogenous matter, part is the resistant albuminoid proteins and part nucleoprotein and nuclein from the intestinal secretions. Thus, a very large per cent (80 to 90) of the nitrogen present in sewage sludge, if not acted on by biological agents, would be present in the form of nucleoproteins and their insoluble intermediate decomposition products.

Sewage sludge, however, has in almost all cases undergone considerable decomposition, particularly in the case of septic tank and Imhoff sludges. Jesse,[15] in studying the gases given off from septic tanks, showed that large quantities of carbon dioxide, carbon monoxide,

methane, nitrogen and hydrogen sulfide were evolved. That large quantities of nitrogen compounds are lost is evidenced by the fact that septic sludge contains only 1 to 2 per cent nitrogen.

Schittenhelm[37] has shown that B. coli splits up nucleic acid into its purine bases and then deammonifies adenine and guanine to give xanthine and hypoxanthine. He also showed[38] that nuclein bases are rapidly lost in putrefying feces, and that if adenine is added to fresh feces after three weeks of putrefaction it has almost entirely disappeared.

Activated sludge is formed under aerobic conditions and the nitrogenous substances are not decomposed by putrefactive processes. The high nitrogen content of the sludge indicates that no great amount of insoluble nitrogen is lost. If we take Hawk's data of 9 per cent total nitrogen in feces and subtract the 32 per cent water soluble nitrogen we get 6.12 per cent nitrogen left. The average analyses of activated sludge in this country and England have shown a total nitrogen content of 5.0 to 6.3 per cent. At first these high results were thought to be due to a large carbonaceous decomposition of the sludge, which left the nitrogen compounds more concentrated. In view of the above considerations this explanation is unnecessary. Besides, the amount of carbonaceous fermentation which goes on must be counterbalanced by an equivalent loss of nitrogen.

In view of the above data it follows that the nitrogen in activated sludge is for the most part in the form of nucleo-proteins and their intermediate decomposition products.

The percentage composition of activated sludge according to present analytical methods for fertilizers is: moisture 10, total nitrogen 5.7, total phosphorus (P_2O_3) 3.9, ether soluble matter (4 hours extraction) 5.0.

	Alkaline permanganate method.	Neutral permanganate method.
Inactive water insoluble nitrogen	3.39	0.67
Active water soluble nitrogen	2.37	5.45
Availability of water insoluble organic nitrogen	44.7	89.0

Such an analysis is arbitrary and gives no indication of the forms of nitrogen present other than their ease of hydrolysis under specified conditions. That these methods are unsatisfactory is universally accepted by scientific men, since they do not compare with vegetation experiments. The above analysis is self-contradictory; according to the alkaline permanganate method activated sludge would be classified as an inferior fertilizer, while the results of the neutral permanganate method

show that it is a good fertilizer. The vegetation experiments demand better understanding of the forms of nitrogen originally present and of their decomposition in explaining the availability of organic nitrogen to the plant.

The forms of nitrogen in activated sludge were studied by extracting known quantities of sludge with nitrogen free water. A summary of results is given below. Extracts 1, 2, 3 and 4 were prepared from Milwaukee activated sludge (4.0 per cent N.) 100 grams were shaken in a shaking machine for 24 hours, settled and filtered until clear and tested as follows:

Buret reaction (+?). Coagulation with heat and acetic acid (—). $CuSO_4$ — $NaHS_2O_2$ test for purines (+). Ammonical $AgNO_3$ test for purines (+). Test for purimidines (—). Test for PO_4^{---} (—). Test for Fe^{++} (—). The biuret reaction was hard to read because the color of the extract was so deep. The percentages of soluble material obtained from the four extracts were so similar that they were averaged:

	Per cent of total.	Per cent of soluble material.	Per cent of total nitrogen.
Sludge dissolved	12.33		
Ammonia nitrogen	.37	2.96	9.15
Organic nitrogen	.97	7.90	24.28
Total nitrogen	1.34	10.86	33.44

It was surprising to find such a large per cent of sludge soluble. Analysis of the sludge moisture before drying showed that an error of only 0.3 of one per cent could be introduced from soluble material in this moisture which the wet sludge contained. Hot water was found to increase solubility to a slight degree as shown below:

	Cold water extract.	Hot water extract.
Soluble matter	10.18 per cent	15.27 per cent
Total nitrogen	1.16 per cent	1.59 per cent
Ammonia nitrogen	.32 per cent	.26 per cent
Organic nitrogen	.84 per cent	1.33 per cent
Per cent of total nitrogen sol.	.29 per cent	37.8 per cent

Sludge as it exists in the purification plant must be made up of insoluble material, for it is constantly stirred with a large excess of water which would dissolve all soluble matter.

The increased solubility of the sludge must be due to a breakdown of the insoluble complexes into soluble compounds in the process of drying. In work described later it was found that acidification of the sludge changes its physical nature and facilitates filtering and drying. A series

of extractions similar to those above, using sludges dried in different ways, acidified and unacidified, are reported in Table 16. The effect of different methods of drying on the solubility of nitrogen is very noticeable.

TABLE 16—RESULTS OF DIFFERENT METHODS OF DRYING.

Number—treatment.	Dried on steam bath.	Centrifuged and dried on cheese cloth in 1 week.	Dried on sand bed 2 months.
Per cent in sludge nitrogen, N	4.4	4.1	4.0
Sludge soluble	6.27	5.42	5.99
Total N_2	1.42	.53	1.54
Ammonia nitrogen	.39	.09	.58
Organic nitrogen	1.03	.44	.96
Per cent total nitrogen in soluble sludge—			
Total nitrogen	32.3	12.9	38.5
Ammonia—nitrogen	8.9	2.2	14.5
Organic—nitrogen	23.4	10.7	24.0

Number—treatment.	Acidified, and extracted.	Acidified, dried and extracted.	Acidified, dried and extracted.
Per cent in sludge nitrogen, N	5.0	5.8	4.6
Sludge soluble	12.51	10.14	16.12
Total N_2	1.20	1.02	1.08
Ammonia nitrogen	.08	.13	.29
Organic nitrogen	1.12	.89	.79
Per cent total nitrogen in soluble sludge—			
Total nitrogen	24.0	17.6	35.0
Ammonia—nitrogen	1.6	2.2	6.3
Organic—nitrogen	22.4	15.4	17.2

The great fluctuation in the ammonia nitrogen is due to the varying amounts of decomposition taking place in the extracts before the extractions were made. The soluble nitrogen is very easily decomposed into ammonia even when preservatives are used. The loss of nitrogen in drying sludges is dependent on the amount of decomposition which takes place. The longer the period, the more bacterial decomposition will take place. The higher solubility of acidified sludge must be due to a hydrolysis of the nitrogenous matter on drying in the presence of acid. As will be shown later the acid-treated sludge is more beneficial to plants than the unacidified sludge.

A study of a hot dilute hyrochloric acid (1 to 3) extract of activated sludge showed on qualitative analysis: Biruet test (+), coagulable proteins (—), reduction of Fehlings soln. (+) phosphate ion (PO_4), (+), ammonia nitrogen (+), Purines (+) and Fe (+).

The reactions of this extract were much stronger than those of the water extract, indicating that more of the nucleo-protein was hydrolyzed.

According to the method for the quantitative determination of Neuclein nitrogen given on page 253 of Cammidge's "Feces of Children and

Adults," activated sludge was found to contain about one per cent of Neuclein nitrogen, (average of four determinations). These results have little significance since the method is purely arbitrary. In the process of the analysis it was found that a 2.5 per cent alcoholic hydrochloric acid solution would extract 22 per cent of the total nitrogen in the sludge. According to the method this is supposed to be pigment nitrogen. The residue after removing the pigmented and nuclein material was found to contain 20.2 per cent nitrogen.

A sample of activated sludge was treated according to Plimmer "Practical organic and bio-chemistry"[31] for the isolation and identification of purine bases. Small amounts of guanine and xanthine, a considerable amount of adenine, and smaller amounts of hypoxanthine were found and identified. No attempt was made to determine these quantitatively on account of lack of accurate methods.

Since activated sludge nitrogen is principally in the form of nucleoproteins and their decomposition products, we are ready to consider what effect these compounds have on the growth of plants. It has been shown above that in water solution culture[35] the decomposition products are readily assimilated by growing plants and are very beneficial to growth. In pot culture, of course, these may be ammonified and nitrified before the plant takes them up. Proof that nucleic acid is readily nitrified in the soil is given by M. J. Funchess.[9] It has also been shown that nucleic acid is in most cases more easily nitrified than dried blood. That activated sludge has given better results than dried blood is partly because above a certain concentration dried blood is toxic to the plant. But why should it be toxic when much more sludge is beneficial? A more complete explanation rests on the forms of nitrogen which are present before and after hyrolysis of the fertilizers in the soil.

The nitrogen of dried blood is mostly in the form of serum albumin and globulin. These proteins are very easily broken down into their amino acids. A few of these amino acids, leucine, histidine, creatinine and asparagin have been shown to be beneficial[36] to plants in pot culture, but a larger number have been found to have no beneficial effect, and aspartic acid, cistein, guanidine and tyrosine were decidedly harmful. In the soil, amino acids are readily ammonified and nitrified if not applied in too high concetration.[9] That too high concentration of these amino acids prevents ammonification and nitrification and is toxic to the plant, may explain our results. On the one hand, activated sludge gives decomposition products that are themselves beneficial to the plant or may be nitrified; on the other dried blood is more easily hydrolized to give amino acids some of which may be toxic to both nitrification and plant growth.

In view of this there may be a fallacy in comparing such different materials, but from a practical point of view dried blood was chosen as the standard because it is generally accepted as the best form of organic nitrogenous fertilizer. It has been shown that activated sludge when applied on an equivalent nitrogen basis is superior to dried blood as a fertilizer; from a theoretical point of view the above comparison of their chemical constitution explains this fact. But theoretical considerations do not always work when we are dealing with such a multitude of conditions as are found in the soil. Series 6 was started on February 2, 1918 with different types of organic nitrogen as fertilizers, and with these various considerations in mind.

This series was planted in sand culture which was fertilized with the equivalents of 5 tons per acre of dolomitic limestone, 1 ton of acid phosphate, ¼ ton of potassium sulfate, and nitrogenous fertilizer. Data will be found in Table 17.

TABLE 17—RESULTS FROM APPLICATION OF DIFFERENT TYPES OF ORGANIC NITROGEN AS FERTILIZERS, SERIES 6, 1918.

Pot	Fertilizer.	Per cent nitrogen in fertilizer.	Grams application of fertilizer.	Tons per acre fertilizer.	Nitrogen equivalent pounds per acre.	Bushels wheat per acre.	Tons straw per acre.
1	Check	0	0	0	0	3.0	.6
2	Unextracted activated sludge	5.	17.37	1.39	139.0	7.0	1.5
3	Extracted activated sludge	4.6	18.80	1.50	139.0	8.2	2.0
4	Acidified activated sludge	5.8	14.90	1.19	139.0	5.5	2.0
5	Uric acid	33.3	2.60	0.21	139.0	.5	1.6
6	Sodium nucleinate	14.5	5.97	0.48	139.0	10.0	2.7
7	Dried blood	13.9	6.25	0.50	139.0	10.2	2.7
8	Dried egg white	12.4	7.00	0.56	139.0	1.3	1.0
9	*Sodium nitrate	16.45	5.28*	0.46	139.0	4.8	3.7
10	Gluten meal	7.0	10.98	0.88	139.0	5.1	2.0

* One-half of $NaNO_3$ applied February 2, 1918; the rest March 29, 1918.

In this series the application of 0.5 ton of dried blood was taken as the standard. This quantity was decided upon from the results obtained with dried blood on sand in Series 4. The three types of activated sludges used represent a sample of sludge obtained by mixing a number of sludges taken from the Water Survey Experimental Continuous-Flow Plant[4] during July, 1917, and dried on the steam bath, and two samples which were acidified, dried and extracted with gasoline. Because of the prohibitive cost and impossibility of getting adenine, guanine, xanthine or hypoxanthine in quantities for pot culture work, uric acid, which is the nearest to these in chemical structure, was used. The sodium nucleinate was prepared by Kahlbaum. The dried blood was the same used in the series of 1917. The protein of blood is albumin

and globulin; since dried egg albumin (Mercks) is a good example of an easily hydrolysized albumin. it was used to see if toxic effects would be produced. The sodium nitrate was "C.P." The gluten meal represents a vegetable protein and its decomposition products.

The sodium nucleinate and gluten meals at first appeared to retard germination, but in a few weeks the plants regained strength and soon

Figure 7.—First five in Series 6, end of eight weeks.

Figure 8.—Second five in Series 6, end of eight weeks.

the sodium nucleinate showed itself to be very beneficial. Uric acid and egg albumin were very toxic to the young plants and retarded growth for 6 weeks. Then the plants looked better and began to grow as if the toxic effect were disappearing. The sodium nitrate and dried blood cultures were the best from the first, with the activated sludge pots almost equally good. Figures 7 and 8 show pictures of this series after 8 weeks,

when the uric acid and egg albumin cultures were recovering from the toxic effect. At the end of three months these cultures were growing fast and showed prospects of as good results as the others.

The plant growth, in this series, shows that dried blood and sodium nitrate give the best results when they are used at this low concentration. No toxic effect was produced by dried blood. Gluten meal is not equal to dried blood as a fertilizer. The very toxic effect produced at first during the breakdown of egg albumin indicates that these intermediate products are toxic to the plant, but after these products had been further decomposed and nitrified the plants grew with renewed vigor, tho seed production was poor. Uric acid gave similar results. It was not used directly by the plant, as Schreiner[35] has shown purines to be. The results with sodium nucleinate show that its nitrogen is readily available and gives results equal to those of dried blood. Not all the nitrogen in activated sludge is available for immediate use; at first rapid growth of the plant is produced, then there is a slight relapse which is followed by a second healthy growth apparently due to the hydrolysis of more nitrogen to an available form. Activated sludge which had been acidified, dried and extracted gave much better results than the untreated sludge. It has been shown that the amount of grease in activated sludge does not decrease its availability, therefore the acid treatment must have caused a beneficial change in the nature of the sludge.

DEWATERING ACTIVATED SLUDGE.

Activated sludge as it comes from the settling chamber of the purification plant is very liquid, containing 98 to 99 per cent moisture. On further settling, this moisture may be reduced to 95 per cent, which is about the limit to which the water will separate on settling. In some plants the sludge may be sold as a fertilizer in this wet condition; but in most cases the moisture must be removed, for this large bulk of water would make the transportation charges prohibitive. Lowering the moisture of activated sludge to 75 per cent will reduce the volume of sludge 80 per cent. In order to market the sludge as a fertilizer the moisture must be reduced to 10 per cent. This reduction from 75 to 10 per cent moisture is easily and economically accomplished in rotary hot air dryers. The real difficulty comes in removing the 20 per cent moisture between 95 and 75 per cent.

Sand beds were first tried by us in the summer of 1916. The results did not prove satisfactory. In 1917, five sand beds were built which were carefully underdrained and protected from rain by a canvas covering. Even with this protection the sludge clogged the sand and the excess water was removed by evaporation; two months elapsed before

the sludge was in condition to be raked off the beds. This slow drying allowed the sludge to undergo putrefaction which not only lowered its fertilizing value but caused nuisance around the plant from odor and flies. In cold climates beds of this type would be of no value during winter, so this method of drying was abandoned.

For experimental purposes much sludge was dried by using coarse cheese cloth to cover the bottom of a box 2½ feet by 3½ feet having a wire net in the bottom. The water filtered through this much better, but even with coarse cheese cloth the pores of the cloth clogged with the gelatinous sludge and a large proportion of the moisture had to be removed by evaporation. For practical purposes cheese cloth could not be used, but for experimental purposes it was very convenient. Under the very best weather conditions sludge would dry in this way in one week. Cheese cloth bags were also used with some success.

The results of Hatton at Milwaukee, and of the packers in Chicago in filterpressing activated sludge have not been duplicated in this laboratory. A Kelly press was used with canvas, cheese cloth, burlap and wire gauzes of various meshes for filtering materials. In every case the sludge clogged the filtering medium and water could not be forced through the press with 250 pounds pressure. The addition of lime and also sulfuric acid were tried, the lime doing no good and the acid being only slightly beneficial. These results show that the colloids of the sludge stop up any type of filtering material tried, and that pressure induces clogging more rapidly than gravity filtration. Therefore, the experiments that follow, attempt to change the physical properties of sludge to facilitate filtration.

In order to study the effect of excess aeration on the physical properties of activated sludge, a 50-gallon barrel was provided with a 12-inch filtros plate near the bottom and a 4-inch air chamber beneath this plate. The air used was measured with a gas meter and all errors due to sampling and evaporation were accounted for. The sludge used in this experiment was taken from the settling chamber of the continous-flow plant and settled in barrels for 20 minutes, the clear liquid was siphoned off and concentrated sludge placed in the aeration barrel. Samples were taken every 24 hours. (Table 18).

Experiments on the rate of settling were made by placing 500 cc. of the sample in a 500 cc. graduated cylinder and noting the time required for settling to marks indicated on the cylinder. For comparison only the time required for settling to 75 per cent of the volume is recorded in Table 18.

In studying the rate of filtration as aeration proceeded two liters of aerated activated sludge in a 2-liter flask were inverted over a 10-inch

corrugated funnel so that a constant level was obtained in the funnel. The filter paper used was carefully selected. The rate of filtration per 100 cc. was timed with a stop watch, but the accuracy of the data was not sufficient to permit the formulation of an equation expressing the decrease in rate of filtration per 100 cc. Table 18 gives the total time required to filter 1,000 cc. under constant level and complete data on the experiments.

The suspended solids gradually went into solution and the dissolved solids increased. Analysis of the sludge showed that this aerobic decomposition does not cause a loss of nitrogen in the sludge, at least in the first seven days.

TABLE 18—DATA ON AERATION OF ACTIVATED SLUDGE.

	Liters.	Gallons.
Capacity of barrel	168	44.4
Total sludge removed for samples	22½	5.94
Loss by evaporation	19	5.02
Loss by evaporation per 24 hours	2.5	.72
Total loss (sample plus evaporation) per 24 hours	5.0	1.37
Sludge remaining after experiment	126½	33.4

Sample.	Total hours areated.	Hours between samples.	Cu. ft. air per 24 hours.	Cu. ft. air per hours per gal.
1	0	0	0	0
2	12	12	210	.487
3	36	24	540	.515
4	60	24	850	.863
5	84	24	675	.710
6	132	48	2(662½)	.718
7	156	24	785	.892
8	180	24	765	.899

Sample.	Time of setting to 75% vol. minutes.	Time of filtration of 1,000 cc. in minutes.	Loss of suspended matter.	Gain in dissolved residue.	Per cent nitrogen in dried sludge.
1	90		0	0	5.95
2	134	34.0		150	6.22
3	136	14.8		162	6.05
4	66	12.2	1,422	146	6.25
5			1,818	168	6.10
6	53	3.1	2,000	256	5.97
7	45	4.2	1.896	344	5.99
8	188	4.3	2,322	496	5.30*

* Some loss of nitrogen through stopper.

After 4 to 5 days of aeration, activated sludge filters with much greater rapidity. The loss of suspended solids during 180-hour aeration was 29.15 per cent. This lowering of the suspended solids may explain the increase in rate of filtration shown in the table.

A second experiment similar to the above was carried out using less air. Altho the results were not of the same magnitude they were of the same order and indicated that seven days' aeration would be the practical limit and that the resistance to filtration is materially lowered. The cost of aeration makes this method expensive, while the benefits derived are not great.

Coagulation.—Coagulants to precipitate the sludge and aid in its filtration were tried.

Those used grouped according to their concentration are as follows:

A.	B.	C.
HCl N/1	NaCl saturated	Solids
NaOH N/1	$Ca(OH)_2$ saturated	Kaolin
Na_2SO_4 N/1	$Al_2(SO_4)_3$ 10 per cent.	$CaCO_3$
Na_3PO_4 N/1	Colloidal Fe (prepared according to Taylor)	
NaCl N/1	Aluminium cream	
$CaCl_2$ N/1		
$FeSO_4$ N/1		

The first experiments were made by adding one cc. of solution to 50 cc. of sludge in Nessler tubes and comparing the rate of settling with a check tube to which nothing was added but the sludge, and also the final volume occupied by the sludge when maximum settling had taken place. The results of these studies placed the reagents in the following descending order according to the decrease in efficiency in increasing the rate of settling. HCl N/1, $CaCO_3$ (saturated), $CaCO_3$ solid, $Al_2(SO_4)_3$ 10 per cent, Na_2SO_4 N/1, NaCl (saturated), $Ca(OH)_2$ (saturated), and Kaolin.

NaCl N/1, $CaCl_2$ (saturated), $FeSO_4$, colloidal iron, aluminum cream, and NaOH N/1 gave no better results than the blank determination. Sodium phosphate (Na_3PO_4) hindered sedimentation.

Best results were obtained with acid and with solid pulverized limestone, the rate of settling being proportional to the amounts added. With acid the sludge first settles, and then if sufficient acid has been added to approach neutrality or acidify to methyl orange, Carbondioxide gas is liberated and the sludge is lifted to the top of the container. Repeated experiments showed that on standing 24 hours the acidified sludge occupied about one-third the volume of that occupied by untreated sludge, although it would be on the surface while the untreated remained on the bottom of the vessel. Large amounts of $CaCO_3$ were found to be as efficient as acid, but these quantities were not practical.

Experiments were next made on the rate of filtration, using HCl and powdered $CaCO_3$. These experiments showed that if the sludge is made acid to methyl orange its rate of filtration is greatly increased, smaller amounts of acid are without appreciable benefit. Ground limestone gave better results with sludge that had been aerated for six days than with fresh sludge. Experiments on the rate of filtration of the

concentrated sludge after treatment with acid showed that the acid sludge filtered 50 per cent faster than sludge treated with $CaCO_3$, while a check filtration experiment using untreated sludge did not filter in 24 hours. At the end of 24 hours the untreated and $CaCO_3$ treated sludge putrefied, while the acid sludge had only a "fishy" odor characteristic of acidified activated sludge. Filtration of acidified, limestone-treated and untreated sludge on Buchner funnels with suction showed that the untreated and the limestone-treated sludge clogged the pores of the paper and did not filter in 24 hours. The acid sludge filtered rapidly and a good cake was formed in less than 12 hours. If activated sludge putrefies it is still more resistant to filtration.

It would be natural to expect acid to solve the problem of filter-pressing this material, but this was not found to be the case with the press used in these experiments. However, a much better cake was formed in the press with the acid sludge than with untreated sludge. Sludge treated with $CaCO_3$ showed no improvement at all.

Centrifuging.—Previous to these experiments the only attempts to use centrifugal force for dewatering activated sludge have been based on the principle of filtration, as in a laundry centrifuge. This is only another way of exerting pressure on filtering material; and, as has been shown above, pressure increased the tendency of the sludge to clog the filtering material. The experiments were not very successful. Those carried on in this laboratory were based on the principle used in a cream separator. The bowl of a small basket type of laboratory centrifuge was made imperforate with a strip of rubber packing. Sludge was allowed to flow in near the center of the bowl which was moving at maximum speed. The heavier material packed against the sides of the bowl and the lighter clear water stood on the inside. As more sludge was added the clear water flowed over the top of the bowl and was caught by the outer stationary casing. When sludge was added until the liquid coming over the edge of the bowl was quite turbid a good cake was usually formed. The following tabulated data show the results obtained at different speeds of revolution of the bowl:

Moisture of sludge used	98.4 per cent
Revolutions per minute of bowl	1100
Cake—Moisture	92.9 per cent

This cake was "runny" but was far better than any obtained from our filter presses.

Revolutions per minute	1500
Cake much more solid, moisture	91.5 per cent

These results were so promising that an old cream separator was purchased and used. The results obtained with higher revolutions per minute are as follows:

Sludge used ..... 99.3 per cent moisture
Cake at 7,000 r.p.m. of bowl..... 88.5 per cent moisture
Cake at 9,000 r.p.m. of bowl..... 86.5 per cent moisture
Cake at 10,500 r.p.m. of bowl..... 85.6 per cent moisture

These cakes were quite solid and retained their shape, standing up to the sides of the centrifuge bowl.

Next a small Tolhurst centrifuge with a 12-inch imperforate bowl was installed at the Experimental Activated Sludge Plant. This machine was designed to run at a speed of 2,200 revolutions per minute, but the pulleys were so modified that the experiments reported below were run at 1,800 revolutions per minute.

For two months this machine was run almost daily in order to determine the best methods of operation and the capacity of the machine. To give an idea of the results, a number of characteristic runs are described below. In all cases the concentration of the sludge used is expressed in percentage volume to which the sludge settled in 30 minutes. This represents the degree of dilution of the sludge better than it can be given in any other way. The sludge was measured into the receiving reservoir of the centrifuge in buckets having a capacity of 7 liters. The general method of operation was to start the centrifuge and allow it to reach its maximum speed, which required a minute, then the sludge was run into the centrifuge bowl near the center from a valve in the sludge reservoir. For a while the effluent which escaped thru an opening in the outer casing would be clear, but later as the sludge cake built up the effluent would become turbid with suspended sludge, and if allowed to run much longer would be of the same consistency as the inflowing sludge. When the sludge began to run out of the effluent pipe the machine was stopped and the sludge cake removed, carefully sampled and its moisture determined.

Data on the first run made after the general method of operation was determined, are as follows:

Percentage to which sludge settled in 30 minutes..... 56
Moisture of sludge used..... 99.19
Liters of sludge used..... 70.0
Pounds of wet cake..... 9.7
Percent moisture in wet cake..... 88.2
Pounds of dry cake..... 1.07

Data on a similar run using sludge which settled to 61 per cent by volume:

Liters of sludge used..... 70
Minutes to run sludge thru machine..... 12
Minutes to clean sludge from bowl..... 8
Pounds of wet cake..... 9.45
Pounds of dry cake..... 1.04
Per cent moisture in wet cake..... 87.8

The cake formed in these tests was quite solid and remained in place on the wall of the centrifuge. The cake was about an inch and a half thick.

A test using more concentrated sludge gave the following results:
Per cent to which sludge settled in 30 minutes..... 95
Liters of sludge used..... 35
Minutes to run sludge thru machine..... 10
Pounds of wet cake..... 9.15
Pounds of dry cake..... 1.07
Percent moisture in wet cake..... 88.9

A test of sludge which settled to 96 per cent was run until the effluent was of the same consistency as the inflowing sludge. The results were better:

Liters of sludge used..... 56
Minutes to run sludge thru machine..... 10
Pounds of wet cake..... 9.35
Pounds of dry cake..... 1.09
Per cent moisture in wet cake..... 85.4

These runs show that the efficiency of the removal of water is not dependent on the original concentration of the sludge. The total time of running was also found to be about the same for both dilute and concentrated sludges, because a dilute sludge may be run through the centrifuge much faster than a concentrated one. In order to get a sludge drier than 88 per cent moisture, sludge must be run through the machine until the effluent is about as concentrated as the influent.

An attempt was made to get a drier cake by adding the sludge rapidly at first and then more slowly, with the following results:

Percentage to which sludge settled in 30 minutes..... 95
Liters of sludge used..... 42
Total time to run sludge thru (minutes)..... 8
Minutes for first four buckets to run thru..... 4
Minutes for last two buckets to run thru..... 4
Per cent moisture in cake..... 89.4

This being unsuccessful, runs were so made that between the additions of sludge the bowl was allowed to spin without the addition of more sludge for a definite period, and then more sludge was added.

One more bucket was added in each case after the effluent showed the presence of sludge.

28 liters of "95 per cent sludge" were put thru the machine in	8¾	minutes
Bowl spun without addition of more sludge	1	minute
7 liters of sludge	3¼	minutes
Bowl spun	1	minute
Total time	14	minutes
Cleaned in	5	minutes
Per cent moisture in cake	88.6	
14 liters of sludge added in	4	minutes
Bowl spun	1	minute
14 liters more	5	minutes
Bowl spun	1	minute
14 liters more	5½	minutes
Bowl spun	½	minute
Cleaned in	5	minutes
Per cent moisture in cake	87.7	

These runs show that intermittent addition of sludge is without advantage. In most of the runs made during the two months, a cake containing about 88 per cent moisture was obtained. If considerable quantities of sludge ran through the machine after the effluent became colored, the best result obtained was a cake containing 84.7 per cent moisture. Sludge acidified with H_2SO_4 was put through and centrifuged in a similar manner with these results:

	Per cent.
Concentration of sludge used	95
35 liters gave sludge cake with moisture of	88
42 liters gave sludge cake with moisture of	84.6
49 liters gave sludge cake with moisture of	82.5
Total time	35 minutes

Better results were obtained by using a 95 per cent acidified sludge; adding the last few buckets of sludge more slowly. Data:

42 liters in	8	minutes
14 liters in	3	minutes
14 liters in	5	minutes
7 liters in	3	minutes
Total 77 liters in	19	minutes
Cleaned in	7	minutes
Total time of run	26	minutes
Per cent moisture in cake	81.95	

The best result was obtained by adding acidified, 85 per cent, sludge with intermittent spinning of the bowl. Data:

49 liters added in	10 minutes
Bowl spun	10 minutes
14 more liters	5 minutes
Bowl spun	5 minutes
14 more liters	10 minutes
Bowl spun	5 minutes

When the bowl was stopped clear water ran down from the cake. This water was removed and the bowl spun 5 minutes longer when 40 cc. more of water were removed.

Total time of run	50 minutes
Per cent moisture in cake	79.15

These results show that with acidified sludge a cake containing 80 to 82 per cent moisture may easily be obtained so that two runs could be made in an hour, whereas with unacidified sludge under the same conditions a cake containing 88 per cent moisture is usually obtained.

Some experiments were made with the speed of revolution of the centrifuge bowl reduced to 1,200 r.p.m.; the centrifugal force at this speed was not sufficient to form a good cake.

The J. P. Devine Company has dried for us 200 pounds of sludge containing 88 per cent moisture and reported that it can be done economically. The cost of acidifying activated sludge as obtained by us in Urbana, with H_2SO_4 at $30 a ton, would be 38 cents per million gallons of sewage treated or 76 cents per ton of dried sludge.

Nasmith and McKay's[27] article, "The fertilizing value of activated sludge," which appeared in print after this thesis was accepted, deserves mention. Carefully conducted garden experiments with radishes, lettuce, beans, beets, tomatoes, carrots, and onions are reported. Activated sludge is compared with manure and with other types of sewage sludge, using an equivalent application on each plot of 14.5 tons per acre. The activated sludge used was air dried and had a very low nitrogen content (2.5 per cent nitrogen), evidently due to the loss of nitrogen by bacterial decomposition of the sludge while drying. Application of 14.5 tons of activated sludge with the usual 5 to 6 per cent nitrogen would be a waste of nitrogen, and probably would produce a poorer crop than much less sludge. However, the experiments showed the following percentage increase of production with activated sludge over that with standard barnyard manure.

	Per cent.
Radishes	40.0
Lettuce	103.3
Beans	77.3
Beets	138.0
Late radishes	316.0
Tomatoes	291.0
Carrots	0.0
Onions, Spanish	191.0
Weatherfield	554.0
Danvers yellow globe	87.1

"From these results it is clearly evident that activated sludge is a most valuable fertilizer."

If these results are compared to those in Table 15, there is a striking resemblance to the results on the "wet sludge plot." Data in Table 14 on the results of high applications of activated sludge (5 per cent nitrogen) to wheat, show that the production of grain is materially decreased as the application is increased. However, the growth of straw is increased and it is probable that high applications to truck garden vegetables would not be detrimental, but would greatly stimulate the growth as Nasmith and McKay have reported.

P. Rudnick[33] of the Armour and Company chemical laboratory in criticizing this article suggests that the results published by Bartow and Hatfield[5] in 1916, which showed that activated sludge gave better results proportionally than dried blood, were "contrary to experience and known facts regarding dried blood which is considered the most valuable of commercial organic ammoniates," and that the results were due to the use of sterile sand, sterile dried blood and activated sludge which "teems with proteolytic and nitrifying organisms."

The sludge used in the early experiments reported above was dried 3 to 7 days on a steam bath. It does not seem probable that the nitrifying organisms could withstand this temperature, since the thermal death point of the nitrite organisms is 45°C. for 5 minutes, and that of the nitrate organisms is 55° for 5 minutes. Numerous experiments on activated sludge by Bartow and Hatfield have shown that there are no nitrifiers present as indicated by incubation of standard ammonium sulfate broth. Again, these organisms are very sensitive to acid, and acidified sludge should be sterile, yet acidified sludge has given the best results in pot cultures.

Furthermore, agricultural practice has not shown that dried blood always gives good results. The Agricultural Experiment Station of the University of Illinois has given up the use of dried blood on a number

of experimental farms in the State because it produces retarding effects. These same results have been found by Lipman of California and Fred of Wisconsin. The generally accepted explanation of these results is that since dried blood is so easily ammonified, an accumulation of ammonia is caused which is toxic to nitrobacter. Winogradski and Olelianski[43] have shown that 0.0005 per cent ammonia retards and 0.015 per cent kills nitrobacter. If the growth of nitrobacter is hindered or stopped the accumulation of nitrite is very toxic to the plant.

The toxic effect of dried blood in sand is plainly shown by the experiments reported in Series 5, Figure 3, and Table 11. These results show that dried blood "which is sterile" when planted in "sterile sand" under the conditions of the above experiments will give good results if used in small enough applications, but that larger applications do produce toxic effects.

Series 6 was fertilized with equivalent amounts of nitrogen which would give the best results with dried blood. This series is the only series where the results with dried blood were as good as those from activated sludge. There is no doubt that dried blood in larger quantities causes toxic effect, and the fact still remains that activated sludge under certain conditions gives better results and less toxic effects than dried blood.

If the work is to be continued in this laboratory or elsewhere the following suggestions are offered: allow the fertilizer to decompose for a month or so in the pot or plot before the seeds are planted; treat all pots or plots with an emulsion made from rich gray silt loam in order that all plots will be sure to have the necessary nitrifying organisms present.

SUMMARY.

In the pot culture experiments of 1915, activated sludge was found to be superior to dried blood, gluten meal, and inorganic nitrogenous fertilizer, when applied to sand. In garden experiments of the same year, activated sludge produced a still larger increase in the growth of lettuce and radishes.

Pot experiments using three types of soil fertilized with septic tank sludge, Imhoff sludge, activated sludge and dried blood proved that sewage sludge has a fertilizer value and that activated sludge nitrogen again gave better results than dried blood nitrogen. Experiments on another type of soil showed that 1.5 tons of activated sludge gave the best result in growing sweet corn. Garden experiments in 1916 produced an increase of 71 per cent in weight of cucumbers due to activated sludge.

Sand cultures in 1917 showed that, in sand, applications of dried blood greater than one-half to three-quarters of a ton were toxic to wheat. Increasing the application of activated sludge on gray silt loam did not prove toxic to the growth of foliage, tho in applications greater than one to two tons, it decreased the yield of grain.

The application of sewage sludges the second year to yellow silt loam did not prove toxic but did not give as large a yield as was obtained the year before. The addition of phosphorus to the white silt loam proved that phosphorus was the limiting element in this series; activated sludge being the only fertilizer which produced a better yield than the check pot.

Garden experiments in 1917 proved that both wet and dried activated sludges were valuable as fertilizers, the wet sludge giving better results.

Nitrogen in activated sludge is present largely in the form of nucleo-protein nitrogen and its hydrolytic products, which have been shown to be beneficial to plant growth. Pot culture experiments show that sodium nucleinate is readily available to the plant and that uric acid, tho it is at first toxic, in time is nitrified or decomposed so that its nitrogen is also available. Egg albumin was highly toxic to young wheat plants, showing that the intermediate hydrolytic products of albumin are toxic. Activated sludge which had been acidified gave much better results than unacidified sludge. This is probably due to some hydrolysis taking place in drying the acidified sludge.

Good results are obtained with dried blood if the application is in small enough amounts that the concentration of toxic substances is low.

Drying activated sludge on sand beds was found to be impracticable in this climate, and filterpressing under conditions available was not successful. Excess aeration of the sludge proved an aid to filtration but was not economical. Experiments with precipitants showed that sulfuric acid could be used to the best advantage in separating the excess water and increasing the rate of filtration.

Centrifuging activated sludge in an imperforate bowl will produce a good cake having a moisture content of 88 per cent. From sludge acidified with sulfuric acid, a cake of 80 per cent moisture may be obtained. A cake of this type may be economically dried to 10 per cent moisture in hot air dryers.

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VITA.

The writer was born in Jacksonville, Illinois, July 7, 1892, and attended the public schools of that city. He was graduated from the Jacksonville High School in 1910. From 1910 to 1914 he attended Illinois College, where he was graduated from the Conservatory of Music in 1913, and received the degree of Bachelor of Science in Chemistry in 1914. From 1914 to 1918 he has been a student in the Graduate School of the University of Illinois, receiving the degree of Master of Science in 1916. While at the University he was student assistant in physiological chemistry from 1914 to 1915; assistant bacteriologist in the Illinois State Water Survey from 1915 to 1916; assistant chemist in the Survey from 1916 to 1917 and chemist in the Survey from 1917 to 1918. He is a member of Phi Lambda Upsilon and Sigma Xi.